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Design and Fabrication of a Laboratory Model Uniaxial Hot Press

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Abstract

The present article describes salient features of an uniaxial hot press designed and fabricated for laboratory use. **A** simple lever and pulley mechanism using dead weights is employed to load the sample placed in the graphite die heated by induction. The unit is useful for hot pressing **12** mm diameter and **15** mm long compacts in vacuum or inert atmosphere at a maximum temperature of $\sim$ 2200 **K.** Preliminary results on hot pressing of hexagonal BN powder (without binder) are presented.

1 Introduction

Hot pressing, often referred to as pressure sintering, is an important fabrication process for metals and ceramics useful in advanced high temperature applications. Hot pressing results in better mechanical properties and microstructure compared to those obtained by conventional press-and-sinter method.

In hot pressing, pressure and heat are applied simultaneously to the sample (in powder or cold pressed compact form) inside a metal or ceramic die. The sample can be heated directly by resistance or induction heating or indirectly by conduction or convection. The application of stress to the heated sample can be uniaxial or isostatic leading to uniaxial hot pressing (UHP) or hot isostatic pressing (HIP) methods respectively.

The application of HIP to high temperature ceramics is limited due to the difficulties associated with the requirement of a hermetically sealed deformable container to exert isostatic pressure on the sample at high temperatures (typically >2100 K). This problem is not encountered in UHP as it requires regid die and punches to contain ceramic powder or cold pressed compact for pressure application. Graphite is generally used for UHP dies and punches as it has high strength at high temperatures, can be easily machined, offers low friction and is readily available. Additionally, it has electrical properties suitable for induction and resistance heating. For these reasons, UHP is widely employed for pressure sintering of ceramics. However, the major limitations of UHP are the inability to produce complex shapes and pressure limitations imposed by the strength of the die materials.

2 Equipment details

The uniaxial hot press, designed and fabricated in our laboratory, is shown in Fig.1. It consists of a hot press module placed between two mild steel (MS) plates of the press frame work. The plates are separated and bolted in position by two MS supporting rods. The bottom plate is mounted on a movable platform which is fitted to a rigid support at a convenient height for easy operation. The top plate is fitted with a gun metal bush at its center for guiding a ground stainless steel piston rod and a MS bracket with a shear pin to provide a hinged joint for one end of the linkage. The other end of the linkage, the piston rod and a grooved MS pulley are fitted to the lever arm by shear pins so that they are free to rotate about the axis of their respective shear pins. The lever arm is made of two MS flats separated by spacers so as to accommodate the linkage, piston rod and MS pulley whose positions are such that the lever arm has the mechanical advantage of 6. The top plate, along with the lever arm and its attachments, can be swivelled about one of the MS supporting rods for easy installation of the hotpress module within the press frame work. A wire rope, 3 mm in diameter and anchored to the rigid support at one end, is taken round the pulley and a dead weight is hung at its free end. The lever arm is designed to apply a maximum load of 250 kg on the piston rod. A stainless steel pressure pad, fitted at the center of the bottom plate, supports the hot press module and ensures alignment of its axis with that of the piston rod to prevent eccentric loading during hot pressing.

The schematic of the hot press module is shown in Fig 2a. It consists of a cylindrical die and punches machined from die grade graphite rods supplied by Graphite India Ltd. The die is surrounded by a mullite tube and the annular gap between mullite tube and die is packed with carbon fibre wool for thermal insulation. The mullite tube is held concentric with the graphite die by two pyrophyllite cylindrical

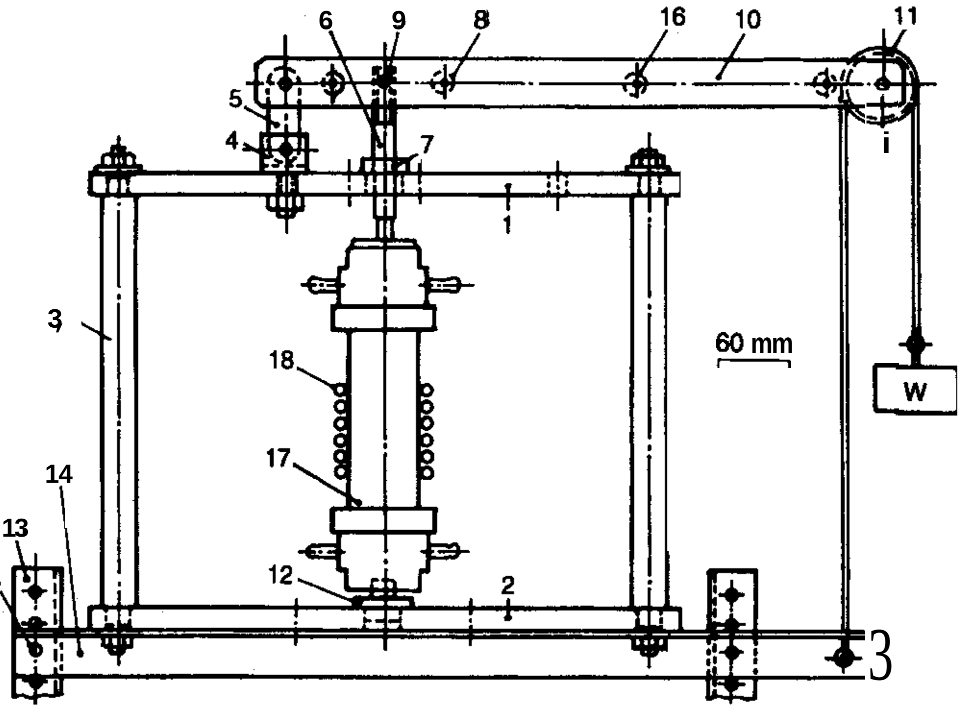


Figure 1: *Schematic of the uniaxial hot press: (1) and **(2)** MS plates, **(3)** supporting rods, (4) bracket, **(5)** linkage, (6) piston rod, (7) gun metal bush, (8) spacers, (9) shear pin, (10) lever arm, (11) pulley, (12) pressure pad, **(13)** rigid support, (14) movable platform, **(15)** supporting bolts, (16) spacer bolts, **(17)** hot press module, (18) RF induction coil, (W) dead weight.*

blocks. The ends of the graphite punches, protruding outside the die, are made larger in diameter to reduce compressive stress on the stressed pyrophyllite components. **A** pyrophyllite punch, located in the central hole of the upper pyrophyllite block, transfers the load to the sample via the upper graphite punch. Pyrophyllite components provide thermal insulation at the free ends of the graphite die and punches. The assembly of graphite die and punches, mullite tube and pyrophyllite components is placed inside a quartz tube that forms the hot press chamber whose ambient can be controlled. The ends of the quartz tube are provided with demountable stainless steel unions for sealing the quartz tube by neoprene rubber O-rings. The unions are water cooled to prevent softening of the O-rings during hot pressing. The pyrophyllite punch is extended into the central hole in the upper union, and the bottom pyrophyllite block is located in a circular recess made in the lower union. This arrangement ensures concentricity of the quartz tube with respect to the die assembly. The lower union has threaded openings for evacuation and introducing the inert gas into the quartz tube. The upper union has a threaded opening for gas vent. The openings for evacuation and gas vent are connected to diaphragm isolation valves and gas inlet is connected to a needle valve. Hot pressing can be carried out either in inert atmosphere or in vacuum by using these valves selectively.

An induction coil, 62 mm ID and 75 mm height and made out of 8 mm OD copper tubing, is positioned outside the quartz tube for induction heating of the graphite die assembly. The coil is fed with the power from 10 kW, 260-450 kHz radio frequency (RF) induction unit (Lepel). An yarn of carbon fibre is helically wound (not shown in figure) over the mullite tube to prevent the tube from shattering due to thermal shock during rapid heating of the graphite die. A stainless steel punch, located in the central hole of the upper union, transfers the compressive load from the piston rod to the pyrophyllite punch.

3 Experimental procedure

The working of the uniaxial hot press was demonstrated by hot pressing 99.9 % pure hexagonal BN powder, having mean particle size of about 10 μm, supplied by Omega Corporation, Bangalore. The following procedure was adopted.

The lower pyrophyllite block, the graphite die and lower graphite punch were assembled. The die cavity was filled with BN powder. The assembly was tapped gently and the powder was hand-pressed using a separate stainless steel punch. This procedure was repeated a few times, after adding some more powder each time, till the die was packed with BN powder to sufficient height of the die cavity. The mullite tube and the upper graphite punch were placed in position and the annular gap between mullite tube and die was packed with carbon wool. The other components of the hot press module were assembled with the quartz tube inserted in the induction coil. The module was placed within the press frame work as shown in Fig.1. A dead weight of 15 kg was placed on the free end of the wire rope. The lever arm was raised and held inclined by inserting 5 mm thick MS spacer between the piston rod and stainless steel punch. Prior to heating, argon gas (IOLAR II grade) was continuously passed through the quartz tube to provide inert atmosphere for the graphite parts of the die assembly. The quantity of gas passing through the chamber was set by the needle valve attached to the bottom union. During this, the diaphragm isolation valve, attached to the bottom union, was kept closed and the isolation or vent valve, attached to the top union, was kept open. The exit gas from the vent valve was bubbled through silicon fluid held in a glass container.

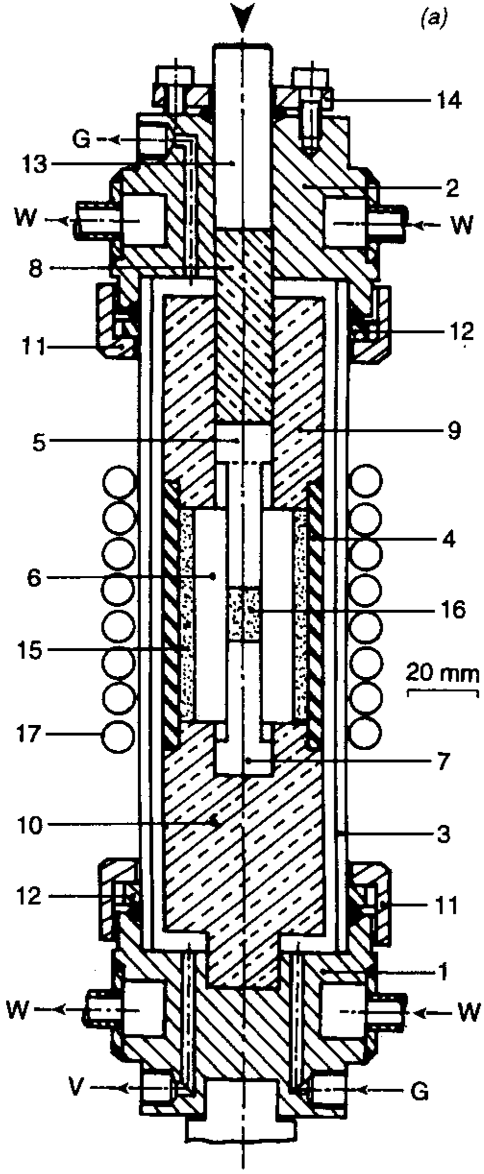
Induction heating of the graphite die assembly was initiated and hot pressing of BN (Fig.2b) was carried out for a duration of 45 minutes. During initial stages, the lever arm shifted down slowly indicating the compaction of the powder. In the final stages, no appreciable movement of the lever arm was noticed. After hot pressing was over and the system attained ambient temperature, the hot press module was removed from the induction coil and the press frame work. Hot pressed BN compact was ejected from the die and its density was determined from the measured volume and weight.

4 Results and discussion

The dead weight of 15 kg, hung at the free end of the wire rope (Fig.1), subjected the wire rope to a tensile force of the same magnitude along its length.. The total downward force acting on the lever arm, at the point where the pulley is fixed, was therefore twice this dead weight (**30** kg). From this force, the pressure exerted by the lever arm, when it was horizontal, on 10 mm diameter BN sample via piston rod and punches was estimated to be 230 bar, after taking into account the self-weights of the lever arm and its attachments.

As hot pressing progressed, the shrinkage of the BN sample displaced the punches and piston rod making the lever arm to shift downwards from its initial inclined position towards horizontal. This movement of the lever caused continuous variation in pressure on the sample being hot pressed. The variation in pressure on BN sample during hot pressing was estimated to be between 220 and 230 bar.The density of the compact was **1.65** gm.cm^{-3} which is **73 %** of the theoretical density of hexagonal **BN** (2.25 gm cm^{-3}). This is better than the density of the hot pressed BN compacts (without sintering aid) reported in the literature'.

Although, the hot press developed in our laboratory has been successfully employed for pressure sintering of BN, there were shortfalls in the experimental work.



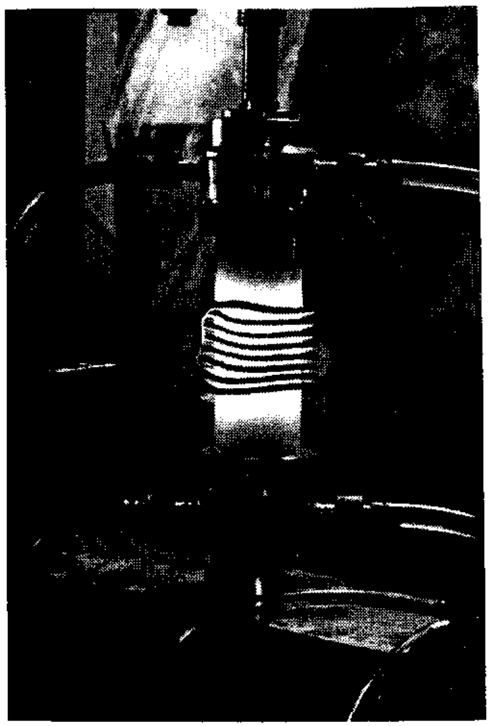


Figure 2: (a) Cross section of the hot press module: (1) and (2) demountable unions, (3) quartz tube, (4) mullite tube, (5) upper graphite punch, (6) graphite die, (7) lower graphite punch, (8) pyrophyllite punch, (9) and (10) pyrophyllite blocks, (11) locknuts, (12) O-ring pressure pads, (13) stainless steel punch, (14) sealing plate, (15) carbon wool, (16) sample, (17) RF induction coil, (W) water, (V) vacuum, (G) inert gas. (b) Hot pressing in progress.

Though we believe from our experience that the temperature was in the region of 2100 K during these experiments, no measurement of temperature was made. Provision is being made in the existing unit to measure the temperature by pyrometry. A high temperature reaction between pyrophyllite and graphite at contact regions was also observed. This is being eliminated by a suitable coating on either graphite or pyrophyllite parts at contact regions so as to make these parts reusable.

5 Conclusions

Application of a simple lever and pulley mechanism, using dead weights, to load the sample during hot pressing is demonstrated. This overcomes the difficulties associated with the use of a hydraulic pump[2] for load application by eliminating manual operation or expensive instrumentation required to maintain constant load on the shrinking sample during hot pressing. The uniaxial hot press has been demonstrated to be a useful laboratory unit by hot pressing hexagonal BN powder.

Acknowledgements

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